December, 1946

The Chemist Analyst

VOLUME 35

NUMBER 4

Published by J. T. BAKER CHEMICAL CO.

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THE CHEMIST-ANALYST

DECEMBER, 1946 VOL. 35, NO. 4

Published by J. T. BAKER CHEMICAL CO., Phillipsburg, New Jersey

Subscription rate, 12 issues $1.00 in U. S. and Possessions. Canada and Foreign $1.50

S. B. WILDRICK, *Editor*

Today's No. 1 job for thinking citizens is to make the American people conscious of the advantages of democracy and free enterprise. We are now the last stronghold of democracy, and unless we make democracy work it may give way to one of the less desirable governmental systems now strongly entrenched in many parts of the world.

To meet this challenge many American business and political leaders are calling for all-out support for the things loyal Americans stand for. They feel it is time that the American people be made conscious of what democracy really means.

Political and industrial leaders who are concerned with the threat to American democracy should find ready allies in all fields of science.

At such a time as this American men of science should not hesitate to offer their full cooperation to those who are leading the way. Few men stand to lose as much if democracy dies—or to gain as much if democracy lives.

If the same amount of energy and the same brilliant thinking as were expended in the development of atomic energy during the war could be used to sell democracy to the world, there would be no doubt of the outcome.

Scientific thinking today is a hundred years ahead of our political and social thinking. The marvelous advances in medicine, physics and chemistry emphasize just how much more progress has been made. Although the scientist has made tremendous progress in improving the welfare of mankind, it is unfortunate that our lack of social progress has caused many of his discoveries to be utilized as implements of destruction.

The men who have solved atomic energy and then used it for war are now applying it to medical and industrial uses, which will make this a better world in which to live. The men who had to produce the terrible ingredients of biological warfare are also the men who are producing the new powerful drugs and germicides that save life.

In a truly democratic world all products of science will be used for the betterment of mankind. If men of science apply themselves as diligently to our social, economic, and political problems, they may be able to offer solutions where others have failed.

Comment on Raithel's "Note on Dimethylglyoxime Reagent"

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and
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There was published recently a short paper by Raithel (*1*), proposing as an aqueous dimethylglyoxime reagent a solution prepared by dissolving a mixture of solid dimethylglyoxime and solid sodium peroxide in water. In view of the vigor with which sodium peroxide and organic material often react, it seemed desirable to check Raithel's suggestion to determine if a hazard existed.

One gram of sodium peroxide, taken from a freshly opened can, and 1 g. of dimethylglyoxime were thoroughly mixed in the dry state. A small amount of water was added from a wash bottle. A violent reaction occurred, flame shot from the beaker and material was projected from it.

This experiment was repeated with larger quantities of water, added all at once. With volumes of water up to about 30 ml., the reaction was violent and flame was produced. With larger volumes, the reaction was not violent but considerable foaming took place; the dimethylglyoxime dissolved, the solution eventually becoming clear with the continued formation of gas bubbles for several days.

In view of this, Raithel's directions should be modified to read (paragraph 5, line 3) "and the mixture dissolved in not less than 50 ml. of water, added all at once, and diluted to 100 ml."

It would appear that the sodium peroxide used by Raithel was not fresh and was probably only sodium hydroxide. What makes this supposition more probable is that Raithel obtained satisfactory results for nickel on Bureau of Standards samples. Our determinations of nickel with a reagent prepared in this manner were unsatisfactory, giving results 0.3 to 0.4 per cent too high owing to contamination of the nickel dimethylglyoxime precipitate by ferric hydroxide. Satisfactory results were obtained when the reagent solution had been allowed to stand about two weeks, at which time the formation of bubbles had ceased and little if any peroxide remained in the solution.

Inasmuch as dimethylglyoxime is a mild reducing agent (as evidenced by its reduction of gold and platinum salts to the metals), it appeared probable that the dimethylglyoxime in Raithel's reagent would be gradually destroyed by oxidation by the peroxide. To settle this question, exactly one gram of dimethylglyoxime was mixed with one gram of sodium peroxide and the mixture was dissolved in 50 ml. of water. After foaming had ceased, the solution was diluted to exactly 100 ml. A 10 ml. aliquot was added to a solution containing an excess of nickel chloride and the solution was made ammoniacal. The precipitate was filtered off and dried in the usual manner. Using the factor 0.806, there was thus found in the freshly prepared reagent 0.09750 and 0.09731 g. of dimethylglyoxime rather than the 0.100 g. which should have been taken. A similar analysis after the solution had stood

three days gave 0.0830 and 0.0830 g. This decomposition is considerably more rapid than that of a solution of dimethylglyoxime in one per cent sodium hydroxide, for which the corresponding figures were: 0.09863 and 0.09863 (freshly prepared) and 0.09797 and 0.09828 (after three days standing).

It would appear that Raithel's method of preparing a dimethylglyoxime solution has little to recommend it.

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Sulfate Determination in Presence of Chromate

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The procedure described by Hall and Hogaboom (*1*) has been modified by using a solution of agar-agar to coagulate the precipitated barium sulfate, as recommended by Bogan and Moyer (*2*).

Agar-Agar Solution. Suspend 0.5 g. of agar-agar, U.S.P., in 50 ml. of cold water and add this to 450 ml. of near boiling water. Stir constantly as the solution is heated to boiling. This solution is effective for several weeks.

Procedure. For bright dip tanks and chrome plating solutions, pipette a 10 ml. sample into a 250 ml. Erlenmeyer flask. Add 5 ml. of concentrated HCl and 15 ml. of ethyl alcohol. If the reaction does not start spontaneously, warm gently. After the chromate has been reduced, add 25 ml. of distilled water and 20 ml. of glacial acetic acid. Then boil for 15 minutes.

Dilute to a volume of 50 ml. and filter while hot through a No. 42 Whatman paper into a 250 ml. beaker. Wash both flask and paper several times with distilled water.

Dilute to 200 ml. and heat to boiling. Add 10 ml. of a 10% solution of barium chloride, dropwise with vigorous stirring. Let settle on the cooler part of the hot plate for about 15 minutes. Then test for completeness of precipitation. If precipitation is complete, add 0.5 – 1.0 ml. of the agar-agar solution, dropwise and stirring for about 10 seconds after each addition. A coarse coagulated precipitate forms, which settles quite readily.

After 1 – 1½ hours filter through a No. 42 paper, scrubbing the beaker. Wash the precipitate on the paper until the chromium has been removed. Then fill the funnel with 1% HCl solution, let drain and wash two or three times with distilled water.

Transfer paper and precipitate to a weighed porcelain crucible. Place the crucible at the entrance of the muffle furnace and burn off the paper. Then ignite for 30 minutes at 850° C., remove, cool in desiccator and weigh the barium sulfate.

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An Aliquot Procedure for Steel Analysis Using Colorimetric Methods

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In steel analysis it is customary to perform a determination of each element on a separate weighed sample. However, when many steels are to be analyzed, considerable time can usually be saved by running a number of these determinations on aliquots of the solution of a single weighed sample of each steel. With the introduction of perchloric acid an opportunity presented itself to use the filtrate of the silica determination for other determinations, and some work has been done in this direction (*6*, *7*). In this paper the silica filtrate is used for the determination of all the commonly occurring elements, except carbon and sulfur, using modifications of colorimetric methods appearing in the literature. Colorimetric methods were chosen because of their speed and simplicity. These methods are capable of giving satisfactory results for routine work on steels containing up to 3.5% Ni, 2.5% Cr, 1.0% Mo, 1.5% Mn, 0.10% P, 0.50% Cu and 0.30% V. A Coleman Universal Spectrophotometer Fluorometer, Model 11, was used for this work; but other instruments should be capable of giving satisfactory results.

Procedure

Determination of Silicon and Preparation of the Solution. Transfer a 2.00 g. portion of the steel to a 250 ml. beaker covered with a watch glass. Add 10 ml. of a mixture consisting of 2 parts nitric acid and 1 part hydrochloric acid. When the reaction has subsided, add 20 ml. of 70% perchloric acid, turn the heat on, evaporate the solution until the heavy fumes of perchloric acid appear and continue fuming for about five minutes. Remove the beaker from the hot plate, hold it over the surface of the water bath for about 5 seconds and then immerse in the cold water. When the sample is cool, carefully rinse off the underside of the watch glass and wash the inside of the beaker with distilled water. Add about 50 ml. of water, stir to dissolve the salts and filter through a 9 cm. No. 41 Whatman or similar paper. The filtrate is received in a 250 ml. Erlenmeyer flask with an accurately placed mark at 200 ml. Wash the silica 7 times with hot 2% perchloric acid. Replace the flask containing the filtrate with the original beaker, wash the paper 7 times with hot 5% hydrochloric acid and twice with water, throwing these washings away. Transfer the filter paper to a porcelain crucible, char, ignite, cool in a desiccator and weigh as SiO_2. Correct for a small blank. Dilute the filtrate to the mark and mix thoroughly.

For those samples which do not require silicon, transfer a 500 mg. portion of drillings to a 50 ml. Erlenmeyer flask provided with an accurately placed mark at 50 ml. and a small lip. Add 5 ml. of dilute nitric acid (380 ml. HNO_3+ 620 ml. H_2O) and 5 ml. of 70% perchloric acid. Cover with a small watch glass and fume as above. Cool rapidly in a water bath, dilute to the mark and mix well.

Manganese (9). Transfer a 1 ml. aliquot to a test tube in a metal rack. Add 10 ml. of a periodate mixture (5.0 g. $Na_2H_3IO_6$,* 100 ml. H_3PO_4,

* Marketed by the G. Frederick Smith Chemical Co. See also: H. H. Willard in "Inorganic Syntheses," Vol. I, 168-171, McGraw-Hill Book Company, Inc., New York, 1939.

900 ml. H_2O). Place a thermometer in the test tube and place the rack in a pan of boiling water. One minute after the temperature has reached 95°C., the tray is removed to a cold water bath. When cool, measure the transmittance at 550 mμ. Subtract 0.01% Mn for each 2% Cr present.

Phosphorus. The method of Hague and Bright (*1*) may be used directly on a 5 ml. aliquot after adjusting the acidity with 10 ml. of sulfuric acid (140 ml. H_2SO_4+2000 ml. H_2O). However, the authors prefer a slight modification using test tubes and requiring no final volume adjustment. Transfer a 2 ml. aliquot to a test tube in a metal rack. Add 4 ml. of dilute sulfuric acid (140 ml. H_2SO_4+2000 ml. H_2O) and then 6 ml. of sodium sulfite solution (10 g./l.). Place a thermometer in the tube and place the tray in a bath of boiling water. Remove the tray from the hot water one minute after the temperature has reached 95°C. Then add 8 ml. of a mixed solution prepared as follows: to 65 ml of water add 25 ml. of ammonium molybdate solution (20 g. ammonium molybdate+300 ml. H_2SO_4 per liter) and 10 ml. of hydrazine sulfate solution (1.5 g./l.). Replace the test tube rack in the boiling water bath and remove to a cold water bath two minutes after the temperature has reached 95°C. Measure the transmittance at 650 mμ. A correction should be made for chromium in samples which contain more than 2% of this element.

Copper (2). *A. Samples containing less than* 0.5% *Ni.* Two 5 ml. aliquots are transferred to 150 ml. beakers. Ten milliliters of citric acid solution (200 g./l.), 10 ml. of ammonium hydroxide (1:1) and 5 ml. of gum arabic solution (10 g. are added to 1 l. of water at 50°C., stir well and add 1 ml. toluene as a preservative) are added to each. To one aliquot add 5 ml. of sodium diethyldithiocarbamate solution (1.0 g./500 ml. water), 15 ml. of water and mix well. To the other aliquot add 20 ml. of water. Measure the transmittance of both solutions at 475 mμ. The corrected reading is obtained by dividing the reading of the colored solution by that of the blank.

B. Samples containing more than 0.5% *Ni.* Two 5 ml. aliquots are transferred to 150 ml. beakers. Add 20 ml of 20% citric acid, 10 ml. of ammonium hydroxide (1:1) and 3 ml. of dimethylglyoxime solution (10 grams dissolved in 650 ml ammonium hydroxide, then diluted to 1 liter). Place the samples in a cold water bath for about 10 minutes and then filter through a medium fritted disc. Add 5 ml. of gum arabic solution to each filtrate and finish as described under *A*, keeping the final volume to 50 ml.

Nickel (5). Pipette 1 ml. of the silica filtrate into a 250 ml. Erlenmeyer flask. Add in succession with mixing between additions: 25 ml. of water, 10 ml. of citric acid solution (200 g./l.), 2 ml. of saturated bromine water, 50 ml. of water and 5 ml. of dimethylglyoxime solution (10 g. dissolved in 650 ml ammonium hydroxide, then diluted to 1 liter). Measure the transmittance at 550 mμ. within 20 minutes.

Chromium (4). Pipette a 5 ml. aliquot into a 100 ml. beaker and add 10 ml. of dilute phosphoric acid (100 ml./l.). Mix well and measure the transmittance at 450 mμ.

Vanadium (8, 10). Pipette two 10 ml. aliquots into 150 ml. beakers. To one aliquot add 4 ml. of a mixture consisting of 175 ml. H_2SO_4 (1:1) + 200 ml. H_3PO_4 +375 ml. water Then add one drop of 30% hydrogen peroxide and shake. To the other aliquot add 4 ml. of a solution containing 10 grams of

ferrous ammonium sulfate dissolved in 140 ml. of the above acid mixture. The corrected transmittance is the quotient of the two readings.

Molybdenum (3). Pipette a 2 ml. aliquot into a 150 ml. beaker, add 15 ml. of dilute perchloric acid (1 $HClO_4$–70% : 2 H_2O) and 5 ml. of 5% sodium thiocyanate solution. Shake well. Then add 10 ml. of stannous chloride solution (dissolve 250 g. $SnCl_2.2H_2O$ in 200 ml. HCl, then add 800 ml. water and a few pieces of mossy tin). Mix well and allow to stand 5 minutes before measuring the transmittance at 465 mμ. Subtract 0.01% Mo for each 2% Cr present. The sodium thiocyanate and stannous chloride solutions should be filtered before use.

Notes

Sufficient fuming and rapid cooling are necessary to avoid low results for chromium. However, to prevent breakage, a procedure similar to that outlined should be followed. Placing the hot beaker on a cool metal plate for 2-3 seconds before holding it over the cold water surface will practically eliminate breakage.

The standardization curves should be prepared preferably from National Bureau of Standards samples run under the same conditions. A set of solutions of these standard samples should be kept on hand for continuous checking.

Matched 19 mm. test tubes were used for measuring the transmittancies. It was found desirable to use six matched cells for each element and to divide the usual range among the six cells. This reduces contamination due to slow drainage when going from a high value to a low value and *vice versa*.

All volumes should be accurately measured and devices such as the Machlett auto-pipette should be used wherever feasible.

In the test tube method for phosphorus, the additions should be made down the center of the tubes as an aid to uniform mixing. Placing a No. 3 one-hole stopper over the burette tip will aid in this centering.

In the method for copper it will be found that the blanks on all plain carbon steels are very nearly the same. This will reduce the number of blanks to be run.

The nickel color is most stable in the *p*H range 8.75–9.15. At higher *p*H the color increases in intensity quite rapidly, and at lower *p*H there is a tendency toward fading. The *p*H can be adjusted quite easily by varying the strength of the ammonia or citric acid. In any event it is best to make up only as many samples as can be run in the next 15–20 minutes.

The molybdenum color is quite stable, fading only to the extent of 0.01–0.03% Mo in three hours, depending on the percentage present.

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An Iodide Method for the Titrimetric Determination of Iodides and Cyanides

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It has been shown (*1*, *2*) that silver nitrate can be titrated with potassium iodide solution with ceric ammonium sulfate and starch as indicator. Reber and McNabb (*3*) have applied this reaction to the determination of chlorides and bromides by adding excess standard silver nitrate solution and, after filtering the precipitate of silver halides, titrating the excess silver nitrate with standard potassium iodide solution.

The purpose of the present work was to apply this method to the determination of iodides and of cyanides, and to compare the results with those obtained by the Volhard method (*4*).

Preparation of Solutions

Silver Nitrate. A 0.1 *N* solution standardized against pure sodium chloride by the Mohr method (*5*).

Potassium Iodide. A 0.1 *N* solution standardized against the silver nitrate solution with ceric ammonium sulfate–starch as indicator (*1*, *2*).

Ceric Ammonium Sulfate. An approximately 0.1 *N* solution.

Starch. A solution containing 0.5 gram of potato starch per 100 ml., made in the usual manner.

Determination of Iodides and Cyanides

Procedure 1. Approximately 0.2 gram of the iodide or 0.1 gram of the cyanide was accurately weighed and dissolved in 50 ml. of water. To the solution 5 ml. of 6 *N* nitric acid and a measured excess of standard silver nitrate solution were added. The silver cyanide precipitate was filtered and thoroughly washed with water containing dilute nitric acid. Sufficient 6 *N* nitric acid was added to the filtrate from the cyanide and to the iodide suspension to bring the acid concentration within the range of 0.2 to 3 *N*. After the addition of 3 ml. of starch solution and 0.1 ml. of ceric ammonium sulfate solution, the excess of silver nitrate was titrated with standard potassium iodide solution to the appearance of a permanent blue-green color. Blank titrations showed that a correction of 0.1 ml. was necessary.

Procedure 2. (For iodides only.) A sample of about 2 grams was accurately weighed and dissolved in water. Ten milliliters of 6 *N* nitric acid were added and the solution was made up to the mark in a 100-ml. volumetric flask and thoroughly shaken. With this solution 25 ml. of standard silver nitrate solution were titrated as under Procedure 1.

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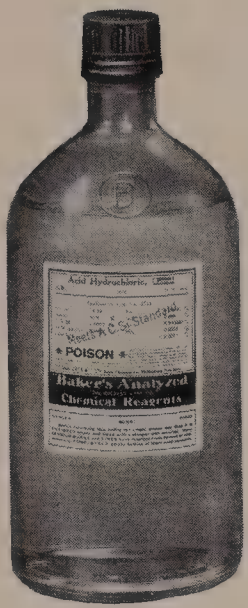

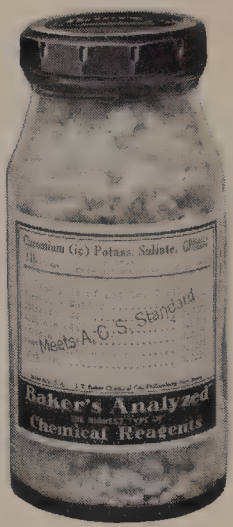

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Results

The results obtained for three iodide salts and for three cyanide salts are shown in Tables I and II, respectively, together with the values given by the Volhard titrimetric method (*4*). The average difference for Procedure 1 for iodides was +0.07 per cent and for Procedure 2 was +0.09 per cent. The average difference for cyanides was 0.00 per cent. Results may be duplicated on the same sample with a precision of about ±0.10 per cent.

TABLE I. RESULTS OBTAINED FOR IODIDES

Sample No.	Salt	Iodide by Procedure 1	Iodide by Procedure 2	Iodide by Volhard Method	Difference Procedure 1	Difference Procedure 2
1	NaI	80.54%	80.52%	80.47%	+0.07%	+0.05%
2	CdI_2	68.40	68.32	68.27	+0.13	+0.05
3	NH_4I	85.80	85.98	85.80	0.00	+0.18

TABLE II. RESULTS OBTAINED FOR CYANIDES

Sample No.	Salt	Cyanide by Procedure 1	Cyanide by Volhard Method	Difference
1	NaCN	49.93%	49.83%	+0.10%
2	KCN	37.41	37.41	0.00
3	KCN	34.51	34.61	−0.10

Discussion

Filtration of the silver cyanide precipitate is necessary because it is more soluble than silver iodide and therefore would slowly react with potassium iodide during the final titration.

The two procedures for iodides give results which check very closely with each other. Procedure 2 has the advantage of requiring only one standard solution.

The method is rapid and easily carried out, especially for iodides where no filtration is necessary. The standard solutions are easily made.

Summary

A titrimetric method has been developed for the determination of iodides and cyanides which consists in precipitation of silver iodide or silver cyanide with excess standard silver nitrate solution and titration of the excess with standard potassium iodide solution with ceric sulfate-starch as indicator. The method gives easily reproducible results which agree well with those given by the Volhard method.

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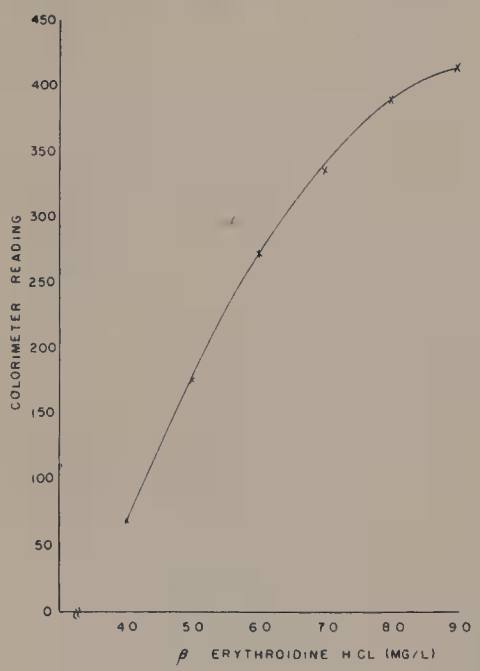
Quantitative Estimation of β-Erythroidine*

SIEGFRIED WOISLAWSKI
School of Medicine and Dentistry, University of Rochester, Rochester, New York

Introduction. In connection with the clinical use of β-erythroidine in patients suffering from infantile paralysis, it was desirable to study the fate of the drug in order to control the drug dosage by chemical tests. We have investigated the use of silicotungstic acid, as applied to the estimation of small amounts of quinine, in the determination of β-erythroidine hydrochloride. The silicotungstic acid procedure was tried because Snell (*1*) had reported that normal urine contains no interfering substances.

Method. In principle the method was that used by Kyker *et al.* (*2*). Because only small amounts of β-erythroidine are excreted in the urine, it was necessary to extract 1 liter of urine for our estimations. The *p*H of the urine was adjusted to 7.0–7.2 by means of $NaHCO_3$ and extracted with chloroform for 4 hours in the continuous extractor of Hershberg and Wolfe (*3*). The chloroform was evaporated on the steam bath, and the residue was dissolved in 10 ml. of 0.03 *N* HCl by placing the flask in a 55° C. bath for 10 minutes with gentle mixing and then in a 100° C. bath for 5 minutes. This procedure facilitated the solution of β-erythroidine hydrochloride. After filtering through glass wool, there was added 0.2 ml. of a silicotungstic acid solution containing 10 g. per 100 ml. The tube was immersed in a boiling water bath for 5 minutes and then cooled in running tap water for 5 minutes. After standing for 5 minutes at room temperature, the turbidity was measured in a Klett-Summerson colorimeter.



Results. In order to have a reference standard, the procedure just described was applied to aqueous solutions of β-erythroidine hydrochloride. The accompanying table and diagram show the relation between the concentration of β-erythroidine hydrochloride and the turbidity as measured by the colorimeter. At the concentration of 30 mg. per liter there is no measurable turbidity.

β-Erythroidine Hydrochloride	Colorimeter Reading of Turbidity
90 mg./l.	415
80	390
70	335
60	272
50	175
40	69
30	-----

* This work was done under a grant-in-aid from the National Foundation for Infantile Paralysis, Inc.

Within the range of 40–60 mg. per liter the relation is practically linear, whereas at higher concentrations the slope of the curve gradually decreases. These values are reproducible and it is therefore possible within these limits to determine β-erythroidine hydrochloride quantitatively in aqueous solutions.

With urine, however, reproducible results were not obtained. This was due to the fact that normal human urine contains substances which give turbidities with silicotungstic acid and thereby prevent a quantitative nephelometric determination of β-erythroidine. No attempt has been made to identify these interfering substances. Reduction with $Na_2S_2O_4$ failed to remove them. ($Na_2S_2O_4$ removes pigments.) This phenomenon has not been previously observed because other workers have used small amounts of urine, for example: 10 ml. by Kyker *et al.* (*2*), 10 ml. by Vedder and Masen (*4*), and 1–2 ml. by Foy and Kondi (*5*).

Discussion. Since β-erythroidine is quickly destroyed in the body, only small amounts of the drug are found in the urine. And, since β-erythroidine silicotungstate is less sensitive than quinine silicotungstate†, large amounts of urine are necessary for the test. Normal urine contains substances that contributed turbidities, which varied from 26 up to as high as 210 units as read on the colorimeter. We cannot recommend silicotungstic acid as a reagent for the quantitative determination of β-erythroidine in urine. We are, therefore, unable to support Snell's statement (*1*) that the reagent reacts with no substance present in normal urine.

We have also investigated the different fractions of the urine to find whether the acidic or basic substances or both interfere with the test. We adjusted the *p*H of the urine to 1.0 by means of concentrated HCl and extracted with chloroform for 4 hours in the continuous extractor; then we adjusted the *p*H of the urine to 7.0–7.2 by means of $NaHCO_3$ and extracted again with chloroform for 4 hours. We readjusted again the *p*H to 1.0 by means of concentrated HCl, this time hydrolyzing the urine by boiling for one-half hour under the reflux. After extracting with chloroform for 4 hours, the urine was adjusted to a *p*H of 7.0–7.2 by means of $NaHCO_3$ and extracted for 4 hours. Both the non-hydrolyzed and the hydrolyzed acidic and basic fractions formed turbidities on addition of silicotungstic acid. At present it is impossible to give any suggestions as to the source of the turbidity.

The author wishes to acknowledge the valuable advice and criticism of Dr. Harold C. Hodge, and he notes with pleasure the assistance given by Dr. R. Plato Schwartz.

† About 25 mg. per liter of quinine will give a turbidity equal to that given by 50 mg. per liter of β-erythroidine.

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On the Reduction of Silver Ferricyanide

MORITZ KOHN
Formerly Professor at the University of Vienna*

It is well known that silver ferrocyanide is oxidized to orange-yellow silver ferricyanide by nitric acid. But the reverse change, the reduction of silver ferricyanide to white silver ferrocyanide, can also easily be brought about. In the presence of silver nitrate, silver ferricyanide is reduced to silver ferrocyanide by hydrogen peroxide at ordinary temperature:

$$2\ Ag_3Fe(CN)_6 + 2\ AgNO_3 + H_2O_2 = 2\ Ag_4Fe(CN)_6 + 2\ HNO_3 + O_2$$

Experiment. To 2.5 ml. of 0.1 *M* potassium ferricyanide add 11.25 ml. of 0.1 *M* silver nitrate and 5 ml. of 3% hydrogen peroxide. After half an hour the precipitate has a very slight yellow color and 15 minutes later it is pure white.

The above reaction is strongly accelerated by reducing the hydrogen-ion concentration. This can be done by adding sodium acetate:

$$2\ Ag_3Fe(CN)_6 + 2\ AgNO_3 + H_2O_2 + 2\ CH_3COONa = 2\ Ag_4Fe(CN)_6 + 2\ NaNO_3 + 2\ CH_3COOH + O_2$$

Experiment. Again mix 2.5 ml. of 0.1 *M* potassium ferricyanide, 11.25 ml. of 0.1 *M* silver nitrate and 5 ml. of 3% hydrogen peroxide. On adding 5 ml. of a 10% solution of sodium acetate to this mixture, the silver ferricyanide is immediately reduced to white silver ferrocyanide.

Silver ferricyanide is described in the literature as an orange-yellow substance. If an excess of silver nitrate is added to a ferricyanide solution and the orange-yellow precipitate is filtered and washed until the filtrate is free from silver, it becomes dirty brownish-yellow, a color which is stable when the substance is stored under water in an amber bottle. On adding silver nitrate, the orange-yellow color is restored.

Pure silver ferricyanide also is attacked by hydrogen peroxide at ordinary temperature. If hydrogen peroxide is added to silver ferricyanide that has been thoroughly washed with water, the substance becomes discolored after several hours and finally acquires a greenish-blue color due to the formation of Prussian blue.

Determination of Free CaO in Commercial Lime

A. C. JANZIG
Water Works Department, Minneapolis, Minn.

Several procedures have been advocated for the determination of free CaO in burnt lime (*1*, *2*, *3*). To these there are certain objections. Where distilled water alone is used to dissolve the sample of lime, high results will be obtained in most cases. And the same is true where no filtration precedes the titration of the solution with standard acid.

* Present address: 536 West 113th Street, New York 25, N. Y.

The method found most suitable for evaluating commercial lime, especially for water softening purposes, is the so-called sugar method. For the past two years it has been used satisfactorily in checking carload shipments of commercial lime. It is both rapid and accurate. Any carbon dioxide contamination of the sample during the process of filtering has amounted to less than five parts per million, which need not be considered in the calculations. Most commercial lime contains some MgO, but it has been found that the presence of even five percent of magnesium does not interfere in the determination.

Procedure. Ten grams of sucrose is placed in a dry 100 ml. glass stoppered volumetric flask, and 100 mg. of the finely ground sample of lime is added on top of the sugar. After inserting the stopper, the sugar and lime are intimately mixed in the dry state. Distilled water, free from CO_2, is added to bring the volume up to 100 ml. Then the flask is stoppered and its contents are thoroughly shaken for 20 to 30 minutes. Allow the insoluble matter to settle and then filter the mixture through a dry folded filter paper (Whatman's No. 12 has been found convenient in this work). Fifty milliliters of the filtrate are titrated with tenth-normal H_2SO_4 or HCl, using phenolphthalein as the indicator. The percentage of CaO is found by multiplying by 5.61 the number of milliliters of acid consumed in the titration.

The distilled water used in the determination should be freed from CO_2 by boiling and then protected from subsequent contamination by atmospheric carbon dioxide.

A control sample containing only the sucrose and distilled water, when filtered and titrated with $N/44$ sodium hydroxide (*4*), will show if any correction is necessary for the presence of carbon dioxide.

Discussion. Sucrose forms with calcium oxide one or more compounds known as calcium saccharates, *e.g.* $C_{12}H_{22}O_{11}$. CaO. A 10% solution of sucrose will dissolve more than fifteen times as much calcium oxide as will pure distilled water. Not only does the sucrose insure more complete solution of the free CaO; it also prevents the solution of other alkaline substances whose presence would tend to give high results.

The preliminary mixing of the sucrose and lime in the dry state prevents caking after the addition of the water.

Filtration of the mixture is necessary. Otherwise insoluble alkaline substances, always present in commercial lime, would consume acid in the titration and thereby give higher results.

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LABORATORY SUGGESTIONS

Strip Solar Heater for Evaporation of Alcoholic Solutions

H. S. STRICKLER
The Endocrine Laboratory, Elizabeth Steel Magee Hospital, Pittsburgh, Penna.

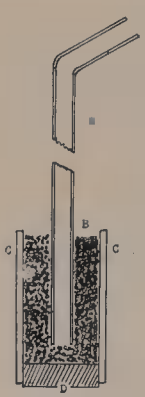
In the evaporation of alcoholic solutions on the water bath, the speed of evaporation is increased and any danger of creeping is minimized by the following device. A three-point strip heater (*1*) is supported, parallel to the top of the water bath, on two ring stands. This permits the simultaneous treatment of several solutions, depending on the number of holes in line on the bath. A piece of asbestos board is cut to size to lie on the top of the strip heater. The rate of heating and the temperature attained by the dry residue is obviously regulated by the 3-point switch and by raising or lowering the heater on its supporting ring stands.

REFERENCE

(1) "Chromalox" Strip Heater, 1500 watt, 3-heat, The E. L. Wiegand Electric Company 7500 Thomas Boulevard, Pittsburgh, Penna.

Device for Dispersing Gases in Liquids

D. F. PETERSEN
Bergstrom Paper Company, Neenah, Wisconsin



For the rapid preparation of chlorine water the pictured device was constructed and found to work very effectively. The gas dispersing medium is glass wool, *B*, which is packed firmly around the gas inlet tube, *A*. The glass wool is held in place by a 2″ length of rubber tubing, *C*, ¾″ or 1″ in diameter. One end of this tubing is plugged with a ¼″ cross section of a rubber stopper, *D*

In certain applications the use of rubber parts might be objectionable, in which case an 18 mm. burette cap or a 2″ length of a ¾″ test tube may be used.

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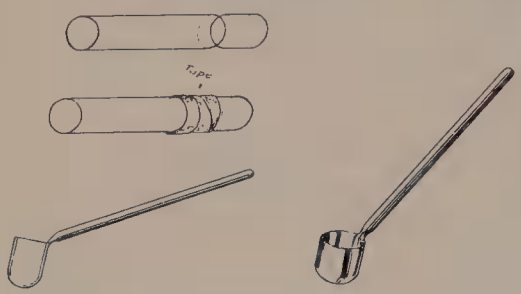
LABORATORY APPARATUS AND CHEMICALS

Dipper for Measuring Chemicals

D. H. MATHESON
Hamilton, Ontario, Canada

In cases where approximate weights of dry chemicals are used repeatedly, measuring with a calibrated glass dipper is convenient. The bowl of the dipper is cut from the bottom of a test tube of suitable size. Weigh the required quantity of chemical, put it in the test tube, shake down with a few light taps and mark the level. It is better if the height of the bowl does not exceed twice the diameter. Cut off the tube at the mark.



For fire-polishing the lip and attaching the handle, the bowl is attached to the remaining piece of the test tube by a turn of adhesive tape as shown, providing a temporary means of holding it. A piece of glass rod, 2-3 mm. diameter, is drawn down to about 1 mm. at one end. The lip of the bowl is fused in the flame, and at the same time the tip of the glass rod is fused and welded on. Anneal well in a smoky flame.

The dipper is conveniently left in the jar of chemicals with which it is to be used, and its capacity marked on the label. The dipper is filled to overflowing and then tapped against the inside of the bottle to shake down the chemical and to shake off the surplus. It is not difficult to make a 5 g. dipper that will measure that amount correct to within 0.1 g.

Gauges for Corks and Rubber Stoppers

EDWIN SCHLESSELMAN
University of Cincinnati, Cincinnati, Ohio

The stockroom of the chemistry department of a college or university generally provides for the needs of students who are working simultaneously in several laboratories. Under such conditions the work of the attendants is greatly expedited if the student places on his requisition the correct number of the cork or rubber stopper needed. But, even though much of the laboratory equipment has standard dimensions, frequently the student cannot determine that number without a gauge of some sort.

From laboratory supply houses can be purchased tapered gauges or measuring cones. Some of these are expensive, but even the cheaper ones soon disappear.

A suitable gauge can be made simply by mounting on a board sets of corks and rubber stoppers, one for each number being carried in stock. A convenient location for such a gauge is at the side of the stockroom window. Its use avoids the bad practice of trying first one cork and then another, a practice which soon leaves many of them contaminated.

An Electrical Heater for Funnels

B. A. FIEKERS
Department of Chemistry, College of the Holy Cross, Worcester, Mass.

In the filtration of hot solutions it is often desirable to employ standard funnels, the stems of which have not been removed or shortened; but a difficulty, arising from the formation of crystals in the stem, is often encountered. Accordingly, the construction and use of an electrical device to provide heat in the stem as well as in the cone of the funnel are here described. The technique for using the device lends the eminent advantage of simplified temperature control.

A 5″ glass funnel with 60° cone and a 4″ stem is used as a form. A semicircle of asbestos paper is cut, moistened, fitted and stuck to the outside of the cone. A spirally wound strip of it is likewise applied to the stem. On drying, this covering adheres to the glass. A heating element, which corresponds to 360 watts and 110 volts, is next wound around the form so as to concentrate the heat in the bottom half of the cone, with some heating in the rest of it and along the stem. For this purpose an 18″ length of No. 22, coil-form, Nichrome wire is used. The element is then packed and covered with an approximately ½″ layer of fireclay from about 1″ below the rim to about 2″ above the tip of the stem. The clay is in turn covered with a few layers of moistened asbestos paper, through which the heating element is led from the lower extremity up to the rim. A short length of the wire at the upper extremity of the unit is also suitably stretched for external connections. This completes the heating unit.

The housing is prepared from a 7″ commercial, sheet iron funnel by cutting its tapered stem at a point which is 1½″ below the cone, and by attaching insulated binding posts or other electrical attachment near the rim. The heating unit is set in an inverted position and the housing is placed over it so as to enclose it completely, except for the rim and the tip of the glass form. A slurry of plaster of Paris is poured into the space between the stems and allowed to set. The assembly is righted again. The extremities of the heating element are connected to the binding posts and the space between the conical parts of the unit and the housing is filled with plaster of Paris. The exposed surface of plaster at the top is carefully pitched so as to drain toward the rim of the housing. When the plaster has set, the glass form is gently twisted by hand and removed. The asbestos covering may come out or be damaged in this operation. It may be replaced with a new asbestos lining that is sealed into the heater with water glass. The heater is then dried

slowly in an oven at low heat or by passing a small electric current through it for some hours. The exposed plaster is then painted. When dry, it is covered with water glass. The outside of the funnel may be protected as desired.

This heater can be set up in a 5″ ring. It will accommodate a 4″ glass funnel snugly, with only rim and tip protruding. The temperature requirement for any given filtration is ascertained easily and accurately. A filter paper is folded, inserted in the funnel and moistened with a cool portion of the solvent to be used in the filtration. The funnel is then covered with a clean, cool watch glass and the current is switched on. When condensate begins to appear on the watch glass, the funnel is ready for use and the current is turned off. It is unnecessary to control the temperature thermostatically or by means of the usual current controls. The appearance of the condensate is a satisfactory temperature indicator.

It is safe to use this technique with most hot organic liquids, because the heating element is not exposed and the inside surface of the heater cannot reach a temperature high enough to ignite inflammable vapors from the liquids that are commonly used. Further, it is very convenient to use such a heater. For the stem provides a heated zone between the receiving vessel and the tip of the filter cone. The possibility of crystallization in the residue on the filter paper is minimized, for the paper is not exposed to the cooler air. The splashing of drops may thus be eliminated, since the tip of the funnel can touch the side of the receiver. Standard funnels may be used and do not have to be shortened. If some crystallization should occur at the very tip of the stem, it is concentrated in a small area and may be removed with a stirring rod, or the funnel may be raised momentarily into the heater to effect re-solution of the crystals. Besides, this apparatus is designed to heat up rapidly, attaining an operating temperature of 100° C. in less than five minutes. It has good heat capacity and keeps the temperature near the boiling point of the solvent sufficiently long for most filtrations. Materials for its construction are generally available. It is simple to construct and operate.

The author is grateful to Arthur T. Martin who helped in the design and has constructed many heaters of this type for use in this laboratory.

A Safe Liquid for Melting-Point Baths

M. MARTIN MAGLIO
Advance Solvents & Chemical Corporation, Jersey City, N. J.

In most laboratories it is customary to employ a bath of concentrated sulfuric acid in making melting-point determinations. The use of this acid is objectionable on account of the inherent danger. Of late some laboratories have adopted one of the "Arochlors"* as a bath liquid. That these liquids possess definite advantages over sulfuric acid is generally conceded.

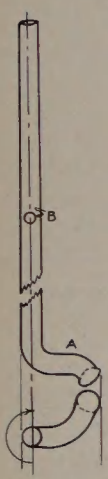
We have found that "HB-40,"* consisting mainly of a mixture of terphenyls, is a very satisfactory liquid for melting-point baths, particularly where temperatures exceeding 300° C. are not desired. We heartily recommend its adoption.

* Marketed by the Monsanto Chemical Company, 30 Rockefeller Plaza, New York City.

A Simple Centrifugal Mixer

KARL E. RAPP
Kentucky Agricultural Experiment Station, Lexington, Ky.

With a minimum of glass blowing experience a very efficient laboratory mixer for liquids may be made from glass tubing by application of the principle of a simple centrifugal pump. This device replaces the stirring rod of the conventional motor- or air-driven laboratory stirrer.



Using 6 mm. glass tubing (this size is adaptable to the chucks of most laboratory motors or is conveniently coupled to the shaft by means of rubber tubing), a smooth right angle bend is made, forming arm *A*. This arm is then bent gently back in the direction opposite to the rotation of the motor, as shown in the projected top view (the mixer illustrated is for use with a motor rotating clockwise). Arm *A* is completed by a slight declination and flaring of the end. Finally, a 3-4 mm. hole is blown in the shaft at *B* and its edges are slightly indented.

The intended application of the mixer determines the height of hole *B*. With the hole just below the surface and the arm near the bottom, the mixer may be used to hasten the solution of solids by circulating the less dense solvent into the more concentrated solution below. The same applies in the dispersion of liquids of different densities. If aeration is desired, it may be accomplished by operating the mixer with the hole above the surface.

Restoration of Glass Thermometers

A. BAILEY
University of Washington, Seattle, Wash.

If mercury thermometers are cooled or heated too rapidly, small checks may occur in the glass bulb holding the mercury. The mercury does not leak but the bulb is exceedingly fragile. The application of a layer or two of "Insalute" cement (Central Scientific Co., Catalog J-141, No. 11470) to the bulb and air-drying restores mechanical strength without change of calibration. Such a repair has given several years of satisfactory service and shows no deterioration from organic solvents or temperature changes. Obviously more lag occurs than with the bare bulb. The repair is especially economical for thermometers with a ground glass joint. If too much cement was applied to the bulb to permit entry into the ground joint, the excess can be ground off with an abrasive stone or file. Similar temporary repairs can be made to other glass apparatus until it can be removed from service and repaired by glass blowing.

MISCELLANY

Attachment for Cenco Photelometer

W. S. PLATNER and D. K. MEYER
Water Quality Laboratories, United States Fish and Wildlife Service,
University of Missouri, Columbia, Missouri

The "Cenco Photelometer" has been extensively used for quantitative work despite its mechanical difficulties. Several workers, as well as the manufacturers of these machines, suggest tapping the machine to assist the microammeter needle in reaching the correct position. In older machines the microammeter becomes quite sluggish and the continuous tapping required becomes boresome.

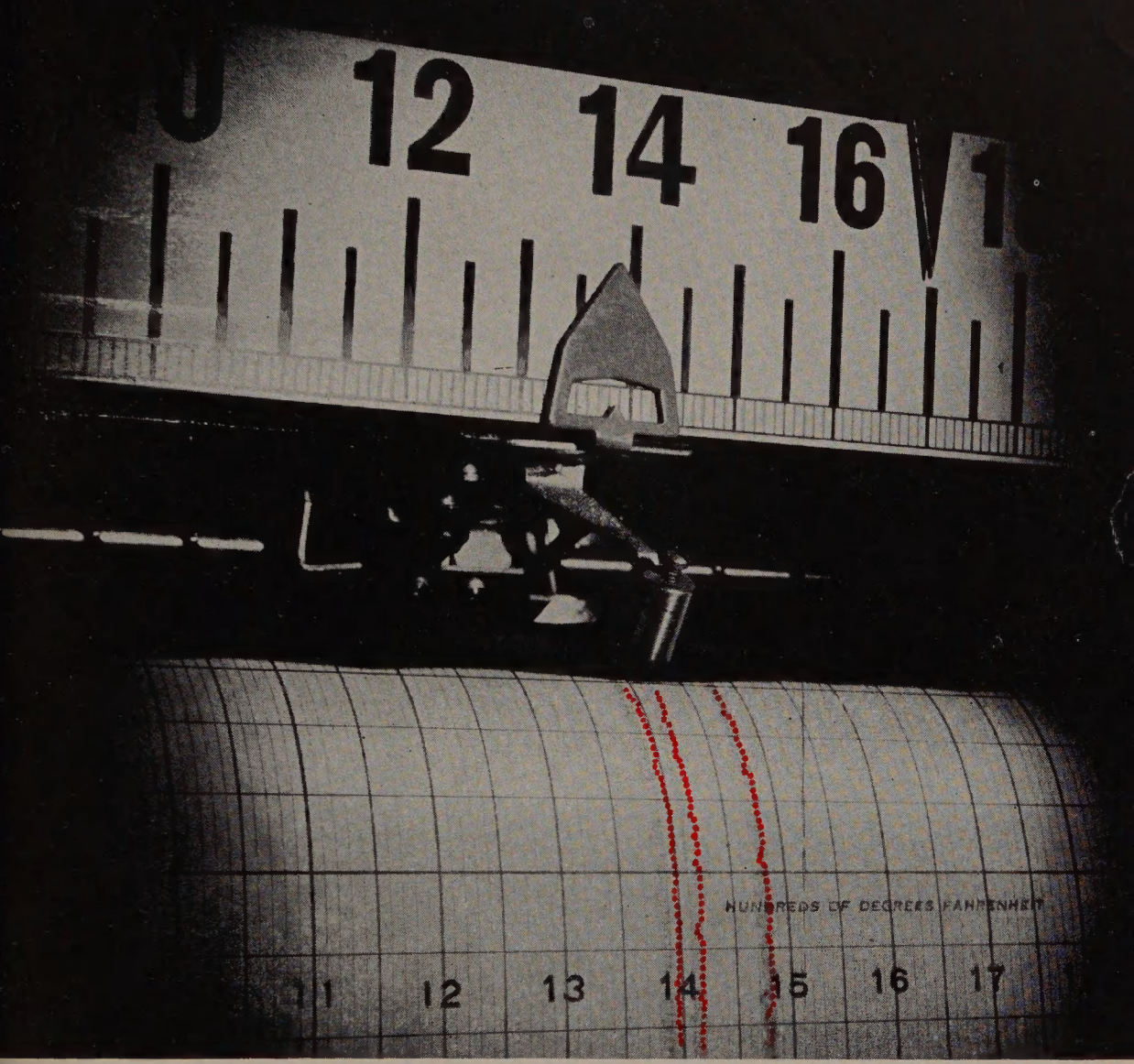
We have found that by attaching an ordinary 6-volt buzzer to the base of a "Cenco Photelometer" and a knife switch to close and open the circuit, easily reproducible readings are obtained. Such an arrangement has been used by the writers in the field and laboratory for more than a year without any apparent injury to the sensitive parts of the machine.

A Substitute for a Laboratory Hood

R. D. COOL
University of Oklahoma, Norman, Oklahoma

The following modifications are suggested with reference to the hood substitute described by Gayle (*Chemist Analyst*, **34**, 96).

It is advantageous to have funnels ranging from one to six inches in diameter so that the maximum draft can be obtained by using the smallest size suitable for the work in progress, particularly when working over a steam bath and a condensate tends to drain down the funnel wall. A long-stem funnel should be used, with the stem bent to form an angle less than ninety degrees. This prevents drainage and dropping of material from the connecting tubing on to the preparation under the improvised hood. One or more traps should be connected between the funnel and filter pump to guard against sucking-back, due to sudden decreases in water pressure, and to protect the pump if it is metallic. "Tygon" has proved superior to rubber for connecting tubing, particularly when removing nitric acid fumes. Sulfuric acid fumes char "Tygon" but still seem to affect it less than they do rubber.



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